STATE OF OHIO
DEPARTMENT OF NATURAL RESOURCES
DIVISION OF GEOLOGICAL SURVEY
Horace R. Collins, Chief

Report of Investigations No. 97

POTENTIAL USE OF OHIO CLAYS IN THE WELL-PLUGGING INDUSTRY

by

Richard W. Carlton

Columbus
1975

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CONTENTS

	Page
Abstract	1
Introduction	1
Properties of well-plugging clays	1
Sample location and description	2
Procedure	2
Results	4
Well-plugging data from Holmes County	4
Commercial sealers	4
Fluid loss	4
Settling data	5
Size analysis	5
Chemical data	5
Mineralogy	5
Statistical data	5
Discussion and recommendations	6
Conclusions	7
References cited	7
Appendix A.–Ohio revised code regulations governing plugging of oil and gas wells in Ohio	8
Appendix B.–Physical data	9

FIGURES

1. Water loss of sample 21 when mixed with various amounts of bentonite ... 4
2. Brine loss of sample 2 when mixed with various amounts of salt-base gel 5

TABLES

1. Sample location and description ... 3
2. Samples arranged in order of increasing brine loss ... 4
3. Clay mineralogy of the $<2\mu$ size fraction ... 6
4. Statistical correlations ... 7

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ABSTRACT

Each year a large number of abandoned oil and gas wells in Ohio are plugged so that hydrocarbons and brine cannot contaminate fresh-water aquifers or land surfaces. Native Ohio clays and rock sediment (combined cuttings and residue from drilling sedimentary rocks) are used extensively for this purpose, but the suitability of much of the material appears to be highly questionable.

Physical and chemical properties of 10 Pleistocene glacial sediments, 12 underclays, and 8 shale and mudstone samples from sites throughout the oil- and gas-producing counties of Ohio were analyzed in an attempt to locate suitable well-plugging material.

Fluid-loss testing was used as the main criterion for determining the well-plugging potential of the samples. Results indicate that determination of the silt content and the settling characteristics of a potential well-plugging material can be useful in approximating fluid loss and can be performed easily in the field. Nine out of 10 of the Pleistocene glacial samples investigated had lower fluid losses than the sample used as a standard.

INTRODUCTION

Each year a large number of abandoned oil and gas wells in Ohio are plugged. The primary purpose of plugging these wells is to isolate the fluids of permeable strata, to permanently prevent the fluids from contaminating one another, and to prevent possible contamination of surface water and land by escape of brine, oil, and gas from the well.

The specific methods and requirements for plugging abandoned oil and gas wells are different in each state. However, as a basic practice it is generally required that productive zones and fresh-water-bearing strata be sealed off from the well bore with cement plugs and that intervals between cement plugs be filled with an approved mud-laden fluid. The Ohio Revised Code (sections 1509.15 and 1509.16) specifies the procedure to follow for plugging oil and gas wells in Ohio (Appendix A).

In Ohio, as in other states, dry rotary-drilled holes are generally plugged with cement and drilling mud; however, drilling mud is not used in holes made by cable-tool methods, nor is it readily available to rotary-drilled holes which are put into production and subsequently abandoned. For holes in which drilling mud is not used or is not available, native Ohio clays, rock sediment, and limestone screenings, generally in combination with cement plugs, have been used.

Although native Ohio clays and rock sediment are used for plugging wells, the suitability of much of the material appears to be highly questionable. At the present time, because of the scarcity of clay in some areas, limestone screenings are being used to plug wells. One sample of this material from Licking County contained 95.2 percent material >2 mm, 3.8 percent material 2 mm to 62.5μ, 0.8 percent material 62.5μ to 2μ, and 0.2 percent material <2μ. Limestone screening is essentially a void filler and has absolutely no water-stopping ability. Furthermore, the clays which are being used are generally chosen for their availability and for the ease with which they can be emplaced in the hole. Unfortunately, many of these clays are either improperly prepared or unsuitable for plugging. For example, clay is generally taken directly from the pit and washed into the well as gravel-sized aggregates which may not disaggregate. The clay may also possess physical and chemical characteristics which make it unsuitable for well-plugging purposes.

A variety of Ohio clays and shales could be utilized as well-plugging material, but, except for a study by the Ohio Division of Mines (Wilson and others, 1944), no systematic approach has been used to evaluate Ohio clays and shales in terms of their well-plugging properties.

The purpose of this paper is to determine some of the physical and chemical properties of Ohio's native clays and shales and to evaluate their potential as well-plugging material.

PROPERTIES OF WELL-PLUGGING CLAYS

The two most important properties of well-plugging mud are the ability to form an impermeable barrier and the possession of a sufficiently high density to displace brine and to neutralize possible high formation-fluid pressures. Low permeability is desirable so that fluids cannot travel from one permeable formation to another. The density of well-plugging mud depends on the specific gravities of the solids and liquids involved in making the mud. Mud density, which can be altered by changing the amount of dry clay added to a known volume of fresh water or brine, generally does not present problems in plugging Ohio oil and gas wells. Some of the major oil- and gas-producing states specify mud viscosities of about 36 (American Petroleum Institute full funnel method) and densities of about 9 lbs/gal.

SAMPLE LOCATION AND DESCRIPTION

The sample material tested was limited to clays, mudstones, and shales which were collected from pits or storage bins of commercial producers or users of clays and shales in Ohio (table 1).

PROCEDURE

The graphic mean, $\frac{\phi_{16} + \phi_{50} + \phi_{84}}{3}$, the inclusive graphic standard deviation, $\frac{\phi_{84} - \phi_{16}}{4.0} + \frac{\phi_{95} - \phi_{05}}{6.6}$, and the percentages of sand plus aggregate, sand, silt, and $<2\mu$ clay were determined. The particle-size distribution of each sample was found by pipette analysis and wet sieving by use of procedures similar to those of Folk (1965).

Fluid loss, the rate at which filtrate is forced from the mud under a specified pressure (Gatlin, 1960), appears to be a good measure of the water impedance of waterborne clays (Rollins, 1969). Results of fluid-loss tests have been used as the main criteria for evaluating well-plugging clays in this investigation.

The amount of water lost from a mud is dependent on a number of variables. In a study of the sealing properties of bentonite suspensions Rollins (1969) found that the water loss from bentonite slurries was least in muds which had the highest percentages of clay and which had adequate exchangeable sodium to form a dispersed structure. It was felt a study along the lines of Rollins' investigation might result in similar conclusions being reached for the montmorillonite-poor clays and shales in this investigation.

Fluid-loss determinations were made with a Baroid filter press (model 300) using compressed nitrogen at 80 psi and Baroid filter paper no. 987. The muds consisted of 200 g of < 25-mesh (0.71-mm) air-dried sample mixed with 200 ml of distilled water. A second group of fluid-loss tests was performed using brine (sp gr 1.165) in place of distilled water. Mixing was accomplished by using a wrist-action shaker and ultrasonic bath.

Samples 2 and 21 were mixed either with a commercial salt-base gel (mostly attapulgite) and brine or a commercial bentonite and distilled water in order to determine whether these products could significantly enhance the fluid-stopping ability of the natural Ohio clays.

The relative degree of settling was determined by mixing 100 ml of distilled water with a 30-g split of air-dried sample of <60 mesh (0.25 mm). Settling characteristics were determined by measuring the height of the mud column after 48 hours of settling in glass tubing of ¾-inch ID. A second set of settling tests was performed using brine (sp gr 1.165) in place of distilled water.

Each sample was x-rayed to determine the clay mineralogy of the $<2\mu$ size fraction. Sample preparation consisted of disaggregation by ultrasonic bath and dispersion with a small amount of sodium hexmetaphosphate; after 4 hours the $<2\mu$ size fraction was pipetted off. The $<2\mu$ suspension was divided into two subsamples. Subsample A was saturated with 1N $MgCl$, and subsample B was saturated with 1N KCl; both subsamples were then centrifuged, and the fluid was decanted. Saturation, centrifugation, and decantation were repeated three times, then each sample was washed three times with distilled water. Smear mounts were made from the K^+- and Mg^{++}-saturated clay pastes.

After glycolation, Mg^{++}-saturated smear mounts were x-rayed at 54 percent relative humidity; K^+-saturated samples were dried at 105°C and heated to 350°C for 4 hours and 550°C for 3 hours, then x-rayed at 0 percent relative humidity.

Peak areas of the glycolated samples were measured with a planimeter. Areas which were measured included the 16.9Å montmorillonite, 14Å vermiculite, 10Å illite, 7Å kaolinite/chlorite, and mixed-layer peaks. Two groups of randomly interstratified mixed-layer clays were distinguished. The most common group consisted of mixed-layer clays which upon heat treatment and/or K^+ saturation collapse to approximately 10Å. Montmorillonite-illite mixed-layer clay and possibly degraded illite are included in this group. A less common group is composed of mixed-layer clays which when K^+ saturated and/or heated retain a broad peak between 10Å and 14Å. Chlorite is always one of the components of this group of mixed-layer clays; these clays may include chlorite-vermiculite, chlorite-montmorillonite, or chlorite-illite.

Cation-exchange capacity was determined on splits ranging from 1.0 to 1.6 g of <60-mesh size. The samples were air dried and transferred to a 40-ml centrifuge tube (oven-dried weights were determined by measuring moisture content at 105°C on separate subsamples). The samples were Na^+ saturated by use of three washes of 25-ml volumes of 1N sodium acetate, then shaken for 5 minutes on a wrist-action shaker. The samples were centrifuged after each sodium acetate wash, and the supernatant was decanted. Following sodium acetate decantation the samples were washed three times with 25-ml volumes of isopropyl alcohol and were centrifuged. The sodium was then extracted with three washes of 25-ml volumes of 1N ammonium acetate having a pH of 7.0.

Extractable Mg^{++}, Ca^{++}, Na^+, and K^+ were determined by transferring 0.9 to 1.7 g of air-dried <60-mesh sample split to a 40-ml centrifuge tube and washing the sample three times in 25-ml volumes of 1N ammonium acetate solution at a pH of 7.0. The Mg^{++}, Ca^{++}, Na^+, and K^+ concentrations were determined on an atomic absorption spectrophotometer using standard methods. Because of the solubility of calcite in ammonium acetate (Chapman, 1965), only the 12 calcite-free samples were used in the statistical study of the extractable-cation data. The presence of calcium carbonate was indicated by observing under the binocular microscope the effect of cold dilute HCl on 1- to 2-g portions of each sample.

Exchangeable cations were determined by subtracting soluble salts from sodium acetate-extractable cations; < 60-mesh sample splits ranging from 9.5 to 10.8 g were suspended in 50 ml of distilled and deionized water for the soluble-salt analysis. The suspension was allowed to stand, with occasional shaking, for 24 hours. A Baroid filter press using Baroid filter paper no. 987 and a pressure of 80 psi nitrogen was used to extract the fluid containing the soluble salts.

TABLE 1.–*Sample location and description*

Sample number	Stratigraphic name or location	Stratigraphic assignment	Textural classification[1]	Mining location: County	Mining location: Township	Source of sample
1	Minford	Pleistocene	clay	Jackson	Madison	Cedar Heights Clay Co., Oak Hill
2	just above Brookville coal	Pottsville	sand clay-silt	Lawrence	Decatur	Marquette Cement Mfg. Co., Bear Run clay pit, Pedro; at approximately 50 feet below Vanport limestone
3	Putnam Hill	Allegheny	silty clay	Holmes	Hardy	Orrville Tile Co.; mined near Millersburg, by Holmes Limestone Co.
4	unknown	Allegheny?	clayey silt	Tuscarawas	York	Wadsworth Brick Co.; mined 6 miles southwest of Dover
5	unknown	unknown	silty clay	Jackson	Madison	Cedar Heights Clay Co., Oak Hill; No. 2 crop clay from storage bin
6	unnamed	Pleistocene	silty clay	Auglaize	Salem	Sandkuhl Tile Co., Spencerville; Pleistocene glacial clay
7	Lawrence	Allegheny	silty clay	Lawrence	Elizabeth	BMI, Inc., Pedro; 12/20 mesh, Lower Kittanning underclay
8	Brookville	Allegheny	silty clay	Tuscarawas	York	Bowerston Shale Co., R.D. 7, Newark; No. 4 clay
9	unnamed	Pleistocene	silty clay	Wayne	Green	Orrville Tile Co.; deep clay (10-50 ft) mined at plant location, about 1 mile southwest of Orrville
10	Brookville	Allegheny	silty clay	Stark	Lake	East Ohio Limestone Co., Hartville; mined at plant location, sold as well-plugging clay
11	Anthony	Pottsville	clayey silt	Medina	Wadsworth	General Wadsworth Brick Corp.; mined 1 mile south of Wadsworth
12	Lower Kittanning	Allegheny	sand silt-clay	Jefferson	Saline	F. J. Dando Co., Irondale
13	Logan?	Mississippian	silty clay	Licking	Hanover	Bowerston Shale Co., R.D. 7, Newark; mined near plant location
14	Brookville	Allegheny	silty clay	Tuscarawas	Wayne and Sugar Creek	Belden Brick Co., Sugar Creek; 1:1 mixture of clays mined in Wayne and Sugar Creek Townships
15	just above Brookville coal	Allegheny	clayey silt	Lawrence	Decatur	Marquette Cement Mfg. Co., Bear Run clay pit, Pedro; red clay overlying sample 2
16	unnamed	Pleistocene	sand silt-clay	Hancock	Marion	Hancock Brick & Tile Co., Findlay
17	Brookville	Allegheny	silty clay	Holmes	Hardy	Clark Clay Co., R.D. 4, Millersburg; sold as well-plugging clay
18	Upper Freeport	Allegheny	silty clay	Tuscarawas	Mill	Superior Clay Products, Uhrichsville; No. 7 brown shale from storage bin
19	unnamed	Pleistocene	silty clay	Henry	Napoleon	Napoleon Brick & Tile Works, Napoleon
20	Lower Kittanning	Allegheny	silty clay	Tuscarawas	Mill	Superior Clay Products, Uhrichsville; No. 5 clay from storage bin
21	unknown	unknown	silty clay	Jackson	Madison	Cedar Heights Clay Co., Oak Hill; No. 1 bond clay from storage bin
22	unnamed	Pleistocene	silty clay	Putnam	Greensburg?	Miller Bros. Clay Works, Inc., Ottoville
23	unnamed	Pleistocene	sand clay-silt	Wayne	Green	Orrville Tile Co.; top clay (less than 10 feet) mined at plant location, 1 mile southwest of Orrville
24	unnamed	Pleistocene	clayey silt	Seneca	Hopewell	J. A. Miller Tile Co., Bascom
25	Bedford Shale	Mississippian	silty clay	Delaware	Genoa	O.G.S. collection (sample no. 21-0885A)
26	unnamed	Pleistocene	clay	Paulding	Jackson	Baughman Tile Co., Inc., Paulding
27	unnamed	Pleistocene	silty clay	Darke	Greenville	Darke County Tile Co., Greenville
28	Brookville	Pottsville	clayey silt	Hocking	Falls Gore	General-Hocking Brick Co., Logan; from pit near Logan
29	Meigs Creek	Monongahela	silty clay	Noble	Jefferson	O.G.S. collection (sample no. 61-0629)
30	Upper Washington	Washington	silty clay	Monroe	Benton	O.G.S. collection (sample no. 56-0648)

[1] The textural classification is taken from Krumbein and Sloss (1963, p. 159).

RESULTS

WELL-PLUGGING DATA FROM HOLMES COUNTY

Because no field testing on the relative well-plugging ability of Ohio clays or bentonites has been performed in this study or otherwise, it is difficult to extrapolate the laboratory data to the wide variety of conditions which could exist in an actual well. However, at least two of the samples (10 and 17) used in the study have been sold as well-plugging material (table 1). Sample 17, from Holmes County, has been used extensively in northeastern Ohio as a satisfactory well-plugging clay and is used as the standard for judging the quality of the remaining samples in this investigation (table 2).

COMMERCIAL SEALERS

The best sealers available in Ohio are probably commercial bentonites and salt-base gels used in the oil well drilling industry. Figure 1 shows that water-loss tests using 100 percent bentonite even at a 1:8 clay-to-water ratio result in water loss more than three times as low as the lowest mean water loss (group A, Appendix B) of a natural Ohio clay at a 1:1 clay-to-water ratio. The salt-base gels, although not as impermeable as the bentonite, have a brine loss of about 60 ml/30 min at 80 psi in a 1:4 clay-to-brine mixture (fig. 2) as compared to the 61.4 ml average brine loss (Appendix B) for all the natural clays tested at a 1:1 clay-to-water mixture.

It would be highly impractical and probably unnecessary to require that all abandoned oil and gas wells in Ohio be filled with well-drilling mud and cement. Figure 1 shows that the addition of even a small amount of bentonite can reduce the water loss of a natural clay drastically.

TABLE 2.—*Samples arranged in order of increasing brine loss*

Sample number	Fluid loss (ml/30 min at 80 psi nitrogen)		Sample type[1]
	Brine loss	Water loss	
1	23.7	25.6	P
26	32.3	27.7	P
19	33.7	26.8	P
7	35.8	44.2	U
22	39.7	27.5	P
27	41.0	30.4	P
6	41.2	51.9	P
24	43.6	31.6	P
9	43.8	57.9	P
16	44.4	38.3	P
20	47.4	50.0	U
5	52.0	85.3	U
17[2]	54.5	66.0	U
21	56.4	55.5	U
13	57.6	58.4	S
8	60.5	71.8	U
15	62.1	75.8	S
23	63.4	44.9	P
12	63.6	16.0	U
10	65.4	76.5	U
29	69.1	91.5	S
4	76.8	67.3	U
25	78.7	87.9	S
3	78.8	80.7	U
18	79.9	76.5	S
14	87.4	85.5	U
28	90.3	86.0	U
11	96.7	122.3	S
30	97.3	96.6	S
2	123.5	103.6	S

[1] P, Pleistocene glacial sediment, U, underclay, S, Paleozoic shale or mudstone.
[2] Well-plugging clay from Holmes County.

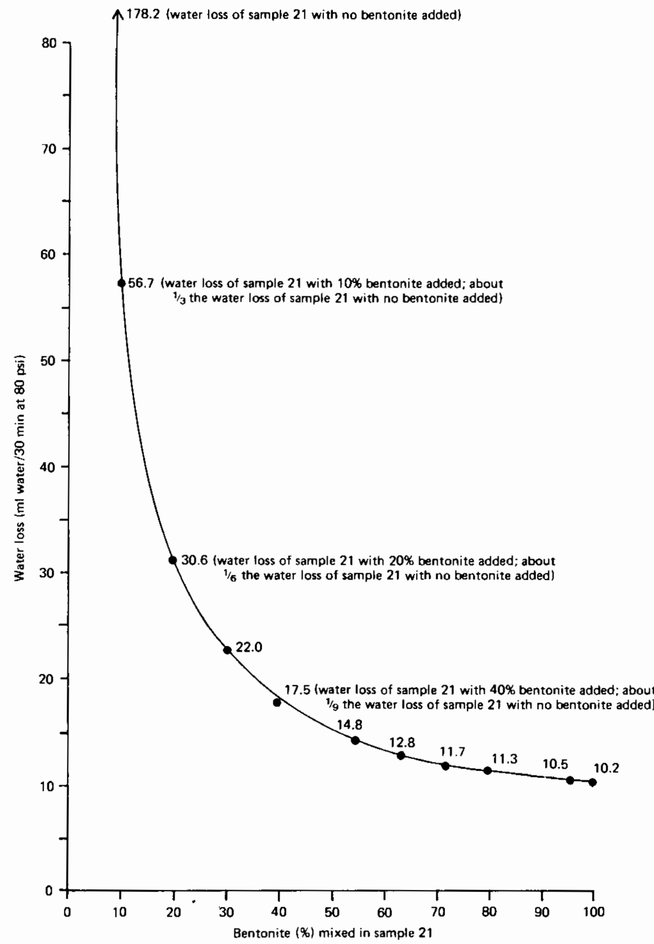

FIGURE 1.—Water loss of sample 21 when mixed with various amounts of bentonite; 50 g solids mixed in 400 ml distilled water and mixed in milk-shake mixer for 2 minutes.

FLUID LOSS

Mean water loss and mean brine loss (Appendix B) were not significantly different, although, given the same conditions, flocculated clay (brine sample) should be more permeable (because of the larger effective grain size) than the same clay in a dispersed state. Apparently the reason mean brine and water losses are nearly the same in this study is that, except for sample 12, which was almost completely dispersed, various stages of flocculation occurred in the distilled water-loss tests. If all the samples had been as well dispersed as sample 12 was in the water-loss tests, then mean water loss would certainly have been lower than mean brine loss in every case.

Nine of the 13 samples with lowest brine loss are Pleistocene glacial sediments (table 2). The mean water and brine losses of the Pleistocene glacial sediments (Appendix B, group A) are much lower than the mean water and brine losses of the underclays (group B) and of the Upper Paleozoic shales and mudstones (group C).

SETTLING DATA

Settling data in Appendix B indicate that on the average a clay slurry will settle to a lower position in a well containing brine than in a well containing fresh water. Too much settling could defeat the purpose of adding the well-plugging mud; to be effective the mud must infiltrate the pores of permeable strata along the hole wall.

The average percent of total fluid column clear after a 48-hour settling period was 44.8 percent in brine and 35.0 percent in distilled water. These values were obtained when using slurries of approximately a 1:3 clay-to-fluid ratio. The least amount of settling as a group occurred in the Pleistocene glacial sediments (group A).

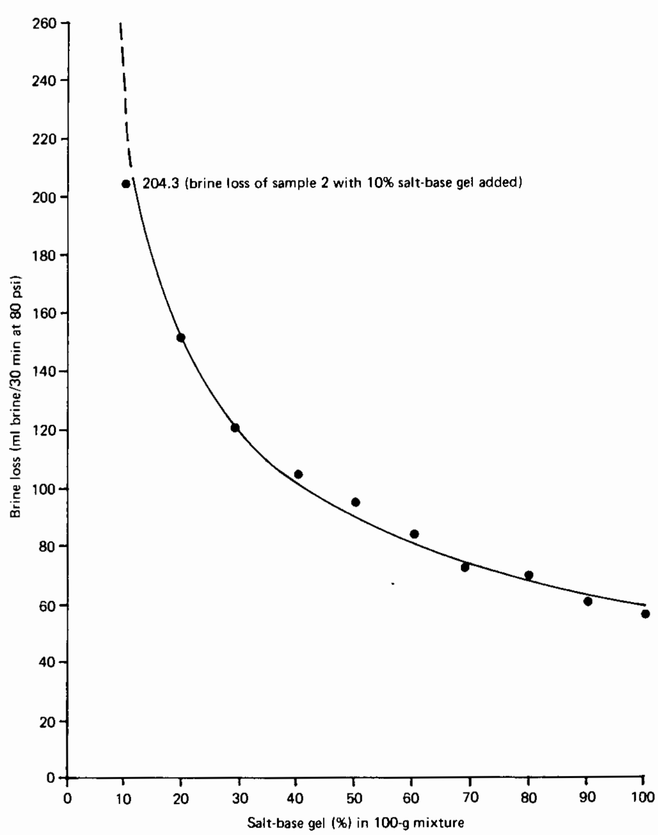

FIGURE 2.–Brine loss of sample 2 when mixed with various amounts of salt-base gel. Clay-to-brine ratio of 100 g to 400 ml, sp gr of brine: 1.126.

SIZE ANALYSIS

The amount of sand plus aggregate, sand, silt, and <2μ clay for the 30 samples is shown in Appendix B. In general, sand and coarser grains are of minor importance in this study. The highest sand content (23.2 percent), in fact, is found in the sample (12) which shows the lowest water loss. As a group the lowest sand-plus-aggregate and silt percentages are found in Pleistocene glacial sediments (Appendix B, group A); sand and clay percentages are highest in the Pleistocene glacial sediments.

Because samples studied are made up almost entirely of silt and clay material, higher silt content generally indicates low clay content and vice versa.

CHEMICAL DATA

Values for soluble Ca^{++} and Mg^{++}, extractable cations, exchangeable Ca^{++} and Mg^{++}, and cation exchange capacity (CEC) are shown in Appendix B. Extractable-cation data are anomalously high in samples which contain large amounts of fine-grained carbonate. Extractable-cation data for only the 12 samples not containing carbonate minerals were used in the statistical study.

In general none of the samples were high in soluble salts, extractable cations, or CEC. Sample 12, with the highest amount of soluble and exchangeable Na^+, had the least amount of flocculation and water loss of any of the samples in distilled water. The remaining samples contained very little Na^+ but apparently enough soluble Ca^{++}, Mg^{++}, and possibly other cations to cause flocculation to various degrees. When mixed with brine, sample 12 did not have an unusually low brine loss, because the high cation content of the brine caused extreme flocculation of the sample.

MINERALOGY

Table 3 lists the clay mineralogy of the <2μ size fraction of the 30 samples included in this study. All the samples contain illite, kaolinite, and mixed-layer clays. Vermiculite occurs in 14 samples, chlorite in significant amounts in 13 samples, and montmorillonite in only 2 samples.

The major differences in mineralogy of the three groups (Pleistocene glacial sediments, underclays, and Upper Paleozoic shales and mudstones) appear to be in the vermiculite and kaolinite contents. The Pleistocene glacial sediments, which as a group have the lowest mean fluid loss and least amount of settling, contain the largest amount of vermiculite and smallest amount of kaolinite.

STATISTICAL DATA

Simple correlations, significant at the 0.02 level or better, exist between water-loss and brine-loss data and percent silt, percent clay, percent sand plus aggregate, and mean phi size (table 4).

The strong correlation between percent silt and water loss supports Rollins' (1969) data in which he found a strong positive correlation (r = +0.826) between silt content and water loss of bentonite suspensions. Correlations of percent sand plus aggregate with brine loss and water loss are significant also and indicate that the amount of aggregate

TABLE 3.—*Clay mineralogy of the <2μ size fraction*[1]

Sample number	Montmorillonite (16.9Å)	Chlorite (7Å)[2]	Vermiculite (14Å)	Illite (10Å)	Kaolinite (7Å)[2]	Mixed-layer[3]
Group A						
1		23		46	16	14
6	11		28	22	8	31[4]
9		23		46	21	9
16		5	32	27	5	30[5]
19		8	29	28	7	28[4]
22		6	40	30	6	17[5]
23		5	41	11	8	34[5]
24		4	29	32	5	29[5]
26		trace	25	30	11	34[5]
27	31		21	15	5	28[5]
mean	4.2	7.4	24.5	28.7	9.2	25.4
Group B						
3				29	56	15
4				17	69	14
5				20	62	18
7			6	7	75	11
8				14	66	20
10			2	28	53	17
12		trace		34	63	4
14				14	69	16
17			2	17	64	17
20			6	7	74	13
21				16	67	16
28				14	60	26
mean			1.3	18.1	64.8	15.6
Group C						
2		12		28	47	13
11		20		44	25	11
13		9	14	34	22	20[4]
15		14		26	37	23[4]
18			14	27	32	27[5]
25		10		44	22	24
29				28	29	44
30		6		35	8	51
mean		8.9	3.5	33.3	27.8	26.6

[1] % of total peak area measured for glycolated samples.
[2] That portion of the 7Å peak assigned to chlorite and kaolinite was determined by measuring on a 1:1 basis the relative peak height of the 002/004 kaolinite/chlorite doublet at approximately 25° 2θ.
[3] The mixed-layer clay is montmorillonite-illite unless otherwise noted; degraded illite may also occur in the mixed-layer clay percentages.
[4] Mixed-layer clay in which one of the components is chlorite (commonly chlorite-vermiculite, chlorite-montmorillonite, or chlorite-illite).
[5] Mixed-layer clay undifferentiated.

present should be considered in choosing well-plugging clays.

Low water loss in general is associated with the more poorly sorted (high standard deviation) samples. This is in agreement with permeability studies which show that in poorly sorted material smaller particles tend to plug voids between large particles (Gatlin, 1960). Because of extreme flocculation of the particles when mixed with brines, no significant correlation exists between the standard deviation (which was determined on the dispersed samples) and brine loss.

Flocculation ratio is the ratio of the amount of <2μ clay in a sample which has not been treated with a dispersant to the amount of <2μ clay in the same sample treated with sodium hexmetaphosphate. The flocculation ratio of the <2μ clay, excluding sample 12, which had a ratio of 0.911, ranged from 0.016 to 0.544. Completely dispersed clay has a ratio of 1.0. The flocculation ratio is related to the amount of soluble Ca^{++} and Mg^{++} in the sample (table 4).

Significant correlations at the 0.01 level exist between settling data and silt content, <2μ clay content, and mean phi size (table 4). Settling in water is also dependent on the degree of flocculation (table 4); in brine the flocculation is complete, and no correlation exists between settling and flocculation.

Statistical analysis of the chemical data indicates that of the chemical tests performed, the cation exchange capacity (CEC) has the most significant relationship with fluid loss. The CEC reflects particle size, mineralogy, and crystallinity of the clay minerals. In a sample such as one of the 30 investigated in this study, CEC should increase with decreasing particle size, decreasing crystallinity, and increasing (relative to chlorite, illite, and kaolinite) montmorillonite or vermiculite content. A more significant negative correlation coefficient probably would have been found between fluid loss and CEC if more than 12 noncarbonate samples (2 from group A, 9 from group B, and 1 from group C) could have been used in determining *r*.

DISCUSSION AND RECOMMENDATIONS

Few if any of the clays and shales investigated in this study will form an impermeable layer as well as commercial bentonites; therefore in areas where there is a serious risk of contaminating aquifers, bentonite or salt-base gel mixed with native Ohio clay should probably be used. If Ohio clays of the type investigated in this report are used alone, clays or mudstones with properties shown in this report to be associated with low fluid loss (particularly material with low silt content, low sand-plus-aggregate content, and high resistance to settling) should be sought out and fluid-loss tests performed, using as a suspending agent the fluid from the well to be plugged. The data presented in this report indicate that Pleistocene glacial sediments (group A) should in most cases make the best well-plugging clays.

In all instances a well-plugging clay should be mixed thoroughly with fresh water (or brine if the well contains brine) before emplacement in the well. Many clays suitable for plugging do not mix readily with water or brine without mechanical agitation. If such clays are dumped into the well dry they tend to bridge, making it impossible to plug the well adequately. The best method is to pump a thick mud slurry to the bottom of the well, where the slurry can displace the less dense brine or water. A less costly, albeit less efficient, method is to pump or dump the slurry in from the top of the well, where it can settle through the column of fluid in the well. If the second method is used the amount of water added to the clay should be kept to a minimum so that mud density will be high. Densities around 9 lbs/gal should be adequate for Ohio's oil and gas wells.

The data in this report are compared to data on the fluid loss of a known Ohio clay (sample 17) which has reportedly been used as a successful well-plugging clay in Ohio. However, the data indicate that underclays, including sample 17, in general do not make the best well-plugging

TABLE 4.—*Statistical correlations*

Dependent variable	Independent variable	Simple correlation coefficient[1]	Equation	Standard error of estimate $(Sy \cdot x)$
Water loss (Y_1)	Percent silt (X_1)	$+0.634_{0.01}$	$Y_1 = 2.41 + 1.58X_1$	20.61
Brine loss (Y_2)	Percent silt (X_1)	$+0.710_{0.01}$	$Y_2 = 4.62 + 1.50X_1$	15.98
Water loss (Y_1)	Percent clay (X_2)	$-0.508_{0.01}$	$Y_1 = 107.94 - 1.07X_2$	22.97
Brine loss (Y_2)	Percent clay (X_2)	$-0.703_{0.01}$	$Y_2 = 115.45 - 1.26X_2$	16.14
Water loss (Y_1)	Mean phi size (X_3)	$-0.444_{0.02}$	$Y_1 = 162.60 - 11.80X_3$	23.89
Brine loss (Y_2)	Mean phi size (X_3)	$-0.719_{0.01}$	$Y_2 = 199.99 - 16.22X_3$	15.76
Water loss (Y_1)	Sand plus aggregate (X_4)	$+0.521_{0.01}$	$Y_1 = 8.49 + 0.29X_4$	12.70
Brine loss (Y_2)	Sand plus aggregate (X_4)	$+0.685_{0.01}$	$Y_2 = -1.06 + 0.45X_4$	10.84
Water loss (Y_1)	Standard deviation (X_5)	$-0.418_{0.05}$	$Y_1 = 135.43 - 22.16X_5$	24.22
Brine loss (Y_2)	Standard deviation (X_5)	-0.161		
Flocculation ratio (X_6)	Soluble $Ca^{++} + Mg^{++}$ (X_7)	$-0.535_{0.01}$	$Y = ab^{X}$	
Settling in water (Y_3)	Percent silt (X_1)	$+0.747_{0.01}$	$Y_3 = -7.50 + 1.12X_1$	10.73
Settling in brine (Y_4)	Percent silt (X_1)	$+0.812_{0.01}$	$Y_4 = 3.59 + 1.09X_1$	8.42
Settling in water (Y_3)	Percent clay (X_2)	$-0.657_{0.01}$	$Y_3 = 71.01 - 0.84X_2$	12.17
Settling in brine (Y_4)	Percent clay (X_2)	$-0.828_{0.01}$	$Y_4 = 85.34 - 0.94X_2$	8.09
Settling in water (Y_3)	Mean phi size (X_3)	$-0.616_{0.01}$	$Y_3 = 119.46 - 9.88X_3$	12.72
Settling in brine (Y_4)	Mean phi size (X_3)	$-0.809_{0.01}$	$Y_4 = 143.89 - 11.59X_3$	8.49
Settling in water (Y_3)	Flocculation ratio (X_6)	$-0.591_{0.01}$	$Y_3 = ab^{X_6}$	
Settling in brine (Y_4)	Flocculation ratio (X_6)	+0.221	$Y_4 = ab^{X_6}$	
Water loss (Y_1)	CEC (X_8)	$-0.347_{0.1}$	$Y_1 = 26.51 - 0.13X_8$	9.47
Brine loss (Y_2)	CEC (X_8)	$-0.359_{0.1}$	$Y_2 = 76.16 - 0.81X_8$	21.18

[1] Subscript indicates level of significance.

clays. Before sample 17 can be completely accepted as an adequate well-plugging clay, much more objective data derived from actual use of this clay in plugging operations should be obtained.

This investigation does not attempt to determine the maximum acceptable fluid loss for a well-plugging clay (although obviously the lower the value the better); until this maximum value can be determined, it is recommended that under normal circumstances sediment with fluid loss not greater than the loss for sample 17 be used as well-plugging material.

CONCLUSIONS

1. Addition of commercial bentonite greatly enhances the fluid-stopping ability of native Ohio clays and shales.
2. Mean water loss and mean brine loss are 62.0 ml/30 min and 61.4 ml/30 min, respectively, for all the naturally occurring samples. When the samples are grouped, however, Pleistocene glacial sediments (group A) have much lower mean fluid-loss values than underclays (group B) and Upper Paleozoic shales and mudstones (group C).
3. Nine out of the 13 samples with lowest brine loss are Pleistocene glacial sediments.
4. The lowest group mean sand-plus-aggregate and silt percentages are found in the Pleistocene glacial sediments (group A).
5. As a group the Pleistocene glacial sediments contain the largest amount of vermiculite and smallest amount of kaolinite.
6. Significant simple correlations exist between fluid loss and percent silt, percent clay, percent sand plus aggregate, and mean phi size.
7. Pleistocene glacial sediments should in most cases make the best well-plugging clays.
8. Well-plugging clays should not be dumped into a well dry or in aggregate form, but should be dumped or pumped in as a slurry.
9. Sediments with fluid loss greater than the fluid loss of sample 17 should not be used in plugging wells.

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APPENDIX A.–OHIO REVISED CODE REGULATIONS GOVERNING PLUGGING OF OIL AND GAS WELLS IN OHIO

1509.15 Method of plugging abandoned wells.

When any well is to be abandoned, it must first be plugged by filling the hole with rock sediment or properly prepared clay to a point above the oil or gas sand or rock formation. There shall then be placed or driven on top of the sediment or clay one or more seasoned wooden plugs or a lead plug as the case may require and such plug or plugs shall be placed or driven in such a manner that the same shall be at the top of the oil, gas, or rock formation, and will prevent the escape of gas or oil and will prevent water or destructive matter entering the oil or gas, sand or rock formation. Such hole shall be filled at least one hundred feet above such plug or plugs or filled to the lowest casing seat with rock sediment or clay and such material used for such filling shall be properly prepared. After the first string of casing has been withdrawn from such well, a wooden plug or iron ball of sufficient size shall be placed upon the casing seat and at least fifty feet of rock sediment or properly prepared clay placed upon such wooden plug or iron ball.

Wells shall be plugged and abandoned in accordance with sections 1509.01 to 1509.19, inclusive, of the Revised Code, and any additional rules and regulations deemed necessary by the chief of the division of oil and gas to obtain proper protection of all formations of economic value.

In the abandonment and plugging of wells located in congested areas, where the plugging method, as outlined in such sections, cannot be applied or, if applied, would be ineffective in carrying out the protection which the law is meant to give, the chief may designate the method of plugging to be used. He may also require the installation of casing and vent pipe to provide additional safety to the surrounding area. The abandonment report shall show the manner in which the well was plugged. (132 v H1. Eff. 2-21-67. 131 v H 234)

1509.16 Plugging and abandonment of well passing through potable water stratum.

If any well has passed through a stratum bearing potable water, it shall, when it is abandoned, be plugged by bridging the hole a minimum of fifty feet below all potable water stratum and filling it to the surface with properly prepared clay or rock sediment. Where there are two or more fresh water strata, a bridge shall be set below the lowest fresh water stratum, and filling shall be continued to a point as specified in this section. (131 v H 234. Eff. 10-15-65)

APPENDIX B.–PHYSICAL DATA

Sample number	Fluid loss of 1:1 clay to fluid suspensions (ml/30 minutes at 80 psi nitrogen)		Total fluid column (%) clear after 48-hour settling period		Flocculation ratio	Grain-size analysis (%)				Statistical measures of size analyses		Chemical data (meq/100-g oven-dried sample)				Carbonate present?
	Water loss	Brine loss	Distilled water-clay	Brine-clay		Sand plus aggregate[1] (>62.5μ)	Sand[2] (>62.5μ)	Silt[2] (62.5μ-3.9μ)	Clay[2] (<2μ)	Mean diameter (φ)	Standard deviation	Soluble Ca^{++} plus Mg^{++}	Exchangeable Ca^{++} plus Mg^{++}	Extractable cations	Cation exchange capacity	
Group A[3]																
1	25.6	23.7	1.8	1.4	0.033	0.2	0.0	1.7	84.6	11.2	2.1	3.2	13.0	73.0	14.0	yes
6	51.9	41.2	26.4	36.5	0.057	16.2	12.8	37.4	41.6	8.6	4.0	1.2	22.2	20.7	23.3	yes
9	57.9	43.8	31.4	26.1	0.027	1.2	0.9	38.5	46.9	9.5	2.9	7.6	7.5	15.8	7.8	yes
16	38.3	44.4	21.8	43.0	0.144	27.2	20.6	34.9	37.7	8.0	4.2	1.0	21.0	22.6	26.2	yes
19	26.8	33.7	13.5	29.6	0.156	21.0	11.9	26.0	53.9	9.3	4.0	0.5	23.9	25.1	35.8	no
22	27.5	39.7	30.8	44.4	0.181	16.4	10.6	29.6	51.7	9.3	4.0	0.4	23.8	24.8	27.1	yes
23	44.9	63.4	50.5	69.4	0.026	21.2	21.2	51.6	22.8	6.8	3.5	0.3	9.8	10.6	13.6	no
24	31.6	43.6	29.2	46.9	0.297	17.8	16.7	41.9	35.7	7.8	3.9	0.5	15.0	16.0	19.6	yes
26	27.7	32.3	8.7	20.1	0.175	11.2	6.4	18.4	66.2	10.4	3.4	1.1	29.9	31.8	41.1	yes
27	30.4	41.0	32.1	51.0	0.125	15.8	7.8	40.6	43.8	8.9	3.8	1.1	25.0	26.7	41.0	yes
mean	36.3	40.7	24.6	36.8	0.122	14.8	10.9	32.1	48.5	9.0	3.6	1.7	19.1	26.7	25.0	
Group B																
3	80.7	78.8	36.4	46.9	0.539	5.2	0.8	44.2	38.6	8.6	2.5	0.9	9.7	11.7	12.6	no
4	67.3	76.8	40.0	46.4	0.376	46.4	8.6	46.1	34.7	7.9	3.2	0.6	8.0	10.3	10.7	no
5	85.3	52.0	39.1	36.7	0.127	21.6	4.5	32.9	50.1	9.3	3.0	0.2	1.5	3.5	13.9	no
7	44.2	35.8	7.7	14.5	0.143	12.6	2.1	23.1	62.7	10.0	3.1	3.4	8.1	12.6	21.6	no
8	71.8	60.5	49.0	46.7	0.042	33.8	8.0	42.8	37.7	8.3	3.3	4.4	7.7	12.9	11.7	no
10	76.5	65.4	43.6	45.5	0.045	40.4	8.4	42.1	38.4	8.2	3.3	6.8	9.0	16.3	9.2	yes
12	16.0	63.6	0.0	44.9	0.911	43.4	23.2	30.9	38.0	7.7	4.0	0.0	3.4	8.9	8.9	no
14	85.5	87.4	53.3	57.8	0.133	48.0	7.9	45.4	36.8	8.0	3.2	1.2	8.3	10.0	10.9	no
17	66.0	54.5	40.5	40.9	0.033	21.6	3.9	36.9	46.7	9.1	3.0	5.9	12.0	18.4	19.5	yes
20	50.0	47.4	49.7	50.2	0.026	24.8	3.6	32.0	54.1	9.1	3.4	1.0	11.3	12.9	12.9	yes
21	55.5	56.4	31.9	51.3	0.028	17.6	6.5	27.9	53.8	9.4	3.3	0.7	10.4	11.9	12.2	no
28	86.0	90.3	28.3	53.9	0.354	33.0	1.3	49.6	34.8	8.1	2.5	0.5	14.0	15.1	17.1	no
mean	65.4	64.1	35.0	44.6	0.230	29.0	6.6	37.8	43.9	8.6	3.2	2.1	8.6	12.0	13.4	
Group C																
2	103.6	123.5	62.9	63.2	0.466	34.2	20.4	49.0	23.4	6.4	3.7	0.5	5.4	6.9	8.9	no
11	122.3	96.7	57.6	53.6	0.043	41.6	10.4	50.9	30.0	7.5	3.1	1.8	4.8	7.3	6.3	yes
13	58.4	57.6	37.1	45.9	0.367	22.4	7.6	44.7	37.4	8.2	3.3	0.9	12.1	13.5	12.6	yes
15	75.8	62.1	43.6	46.9	0.226	14.8	10.0	49.6	32.1	7.8	3.2	1.1	10.0	12.1	13.4	yes
18	76.5	79.9	50.2	56.3	0.350	41.8	16.5	41.4	31.6	7.5	3.6	0.9	14.0	14.7	14.4	yes
25	87.9	78.7	46.2	59.1	0.262	57.6	0.7	36.5	46.6	9.1	2.6	1.1	13.0	14.8	13.4	yes
29	91.5	69.1	33.0	56.0	0.023	33.2	3.6	43.3	39.0	8.4	2.8	0.9	10.2	11.6	32.4	yes
30	96.6	97.3	53.9	59.9	0.544	53.4	6.6	44.9	38.1	8.1	3.4	0.9	38.0	39.7	38.7	yes
mean	89.1	83.1	48.1	55.1	0.285	37.4	9.5	45.0	34.8	7.9	3.2	1.0	13.4	15.1	17.5	
A+B+C mean	62.0	61.4	35.0	44.8	0.209	26.5	8.8	37.8	43.0	8.6	3.3	1.7	13.4	17.7	18.4	

[1] Total material >62.5μ (sand plus aggregate) when disaggregation consisted of slaking 48 hours and stirring with spatula.
[2] Based on complete disaggregation by ultrasonic bath and gentle crushing of aggregate.
[3] *Group A:* Pleistocene glacial and glacially derived sediment; *Group B:* Pennsylvanian or probable Pennsylvanian underclay; *Group C:* Upper Paleozoic shale and mudstone.